

A Sample Inlet System for a Mass Spectrometer

TAUNO MOISIO and ERKKI HONKANEN

Laboratory of the Foundation for Chemical Research, Biochemical Institute, Helsinki, Finland

Gas chromatography combined with mass spectrometry is a valuable tool for identification of volatile substances in microgram quantities. It is usual in mass spectrometric analyses to introduce volatile substances into a reservoir, which is separated from the ionization chamber by a "leak". Direct connection of a "time-of-flight" mass spectrometer to a gas chromatograph^{1,2} makes it possible to scan and record the mass spectrum simultaneously during the gas chromatograph run. These methods are, however, suitable only for lower-boiling compounds. For higher-boiling compounds some methods have been developed^{3,4}, but they are not adaptable to use combined with gas chromatography.

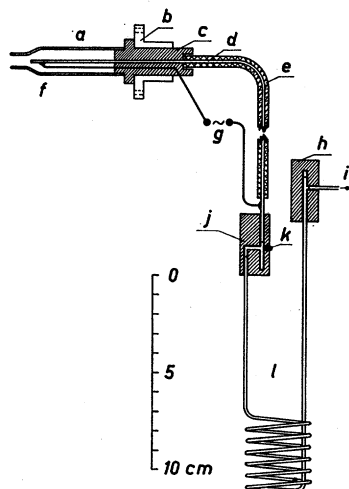


Fig. 1. Diagram of the sample inlet system for a mass spectrometer, a) glass tube, which substitutes for the leak tube; b) brass; c) teflon; d) electrically heated steel capillary; e) thermoisulating; f) analyzer of the mass spectrometer; g) to variable ratio transformer; h) teflon cap; i) to vacuum pump; j) teflon connecting-piece; k) steel tap; l) steel capillary trap.

In the present work a cold-trapping technique and a new inlet system were developed. A conventional mass spectrometer, Modified CEC Model 21-401, was used in the experiments. A schematic drawing of the inlet system is shown in Fig. 1. An electrically heated and thermoisolated steel capillary (i.d. 1.0 mm, o.d. 1.5 mm, length about 100 cm) was connected by Teflon piece to the analyzer of the mass spectrometer, the connection being gas-tight. The other end of this capillary was closed with a Teflon connecting-piece by pushing the capillary end to the bottom of the bore hole with pliers. A special steel capillary trap made from tube of the same diameter (length 50 cm) was then fastened to this connecting-piece after the collection of a compound at -70°C from the gas chromatograph. The other end of the trap was sealed by a Teflon cap and the trap was evacuated at -70°C with a vacuum pump. This end was then closed by pushing the Teflon cap somewhat deeper after which the connection to the analyzer could be opened by pulling the capillary, which leads to the analyzer, outwards. The trap was then brought to a suitable temperature by dipping in a cooling bath so that an adequate vapor pressure was obtained in the ionization chamber. For higher boiling compounds (up to $250-300^{\circ}\text{C}$ at atmospheric pressure) the trap and the connecting-piece were placed in an air thermostat and the capillary was heated electrically (0-12 V, 0-10 A) to about the same temperature.

The weight of sample needed for mass spectrometric analyses by this technique was about $20-100\text{ }\mu\text{g}$ for compounds with molecular weights from 50 to 250, using a scanning time of about 10 min up to $m/e = 250$. The Teflon pieces were found to remain sufficiently gas-tight for several months if the Teflon material was heated at 200°C for some hours before working it.

We thank Professor A. I. Virtanen for his interest in this work. The research has been financed in part by a grant made by the United States Department of Agriculture, Agricultural Research Service.

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Received April 22, 1963.